

SET No: 1EN CHEM SOP
MET-45
REVISION NO. 3
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PAGE 1 OF 4ANALYTICAL METHOD

TITLE: Microwave Assisted Acid Digestion of Aqueous Samples and Extracts

DEPARTMENT: Inorganic - Metals

APPLICATION: Samples may be analyzed by ICP, ICPMS, or GFAA for the following metals:


Al	Ca	Mg	Ag	B
Sb	Cr	Mn	Na	Ti
As	Co	Mo	Tl	Sr
Ba	Cu	Ni	V	Sn
Be	Fe	K	Zn	Bi
Cd	Pb	Se	Li	P
			Si	U

All aqueous matrices apply for use with ICP, ICPMS, or GFAA **except** Total Recoverable metals.

REFERENCE: EPA Manual SW-846, 3rd Edition, Method 3015

PROCEDURE SUMMARY:

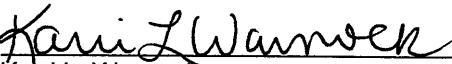
This method prepares samples for dissolved and total metals analysis. Digestion can reduce interferences by organic matter and convert metals to a form that can be determined by instrumentation. A representative 45 mL aqueous sample is digested in 5 mL of concentrated nitric acid (HNO_3) in a PFA digestion vessel for 20 minutes using microwave heating. After the digestion process, the sample is cooled, filtered if necessary, and placed in a clean sample container prior to analysis.

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SAMPLE HANDLING AND PRESERVATION:

All samples are collected in plastic or glass containers. Samples are received by the laboratory acidified to a pH of < 2 with HNO₃ and stored in ambient conditions.

INTERFERENCES:

Many samples that contain organics will produce higher vessel pressures which have the potential to cause venting of the vessels. Venting can result in either loss of samples and/or analytes and must be avoided. In those cases it is recommended to use a Hotblock digestion.

APPARATUS AND MATERIALS:

Microwave - CEM MARS 5
ESP-1500 Plus Pressure Sensor
EST-300 Plus Temperature Sensor
Digestion Vessels-
 Teflon Liners
 Advanced Composite Sleeves
 Vessel Support Modules
 Standard Vessel Covers
vent fittings
brown spacer disks
torque wrench
acid repipettor
pipette: adjustable and fixed
graduated cylinder - 50 mL
Whatman #541 filter paper
D.I. water
Specimen cups

NOTE: The liners and cylinders are nitric acid (HNO₃) and DI water rinsed before use. Be sure the liners are dry before use.

REAGENTS:

Deionized (D.I.) water
Nitric acid (HNO₃): concentrated, redistilled

PROCEDURE:

Quality Control

QC will be contained in each carousel, based on a method blank, laboratory control sample (LCS), and matrix spike (MS) and matrix spike duplicate (MSD) of one per 20 samples, and/or specific instrument needs (ICP, ICPMS, or GFAA).

Sample Preparation

1. WEAR SAFETY GLASSES AND GLOVES.
2. Verify sample Station IDs and LIMS numbers on containers against LIMS worklists.

3. Lay out run in digestion log book for microwave waters. Identify QC samples at frequency stated above.
4. Place Teflon liners in advanced composite sleeves.
5. Aliquot 45 mL of a well-shaken sample into each liner with a graduated cylinder. For method blank and laboratory control sample (LCS) use equal volumes of D.I. water.
6. Spike designated samples using a calibrated pipettor, based on instrument needs (ICP, ICPMS, or GFAA). See SOP MET-56
7. Carefully add 5 mL concentrated, redistilled HNO_3 to each sample vessel. Allow any reaction to be complete before capping any of the vessels.
8. Using the rupture disk installation tool, place a rupture disk into each vent fitting. Screw the Teflon vent fitting onto the threaded stem of the vessel cover. Place a vent fitting with rupture disk on temperature/pressure vessel cover.

NOTE: It is important that the rupture disk be seated flat in the vent fitting. Rupture disks not seated properly will either vent contents under pressure or will result in a substandard run. This will result in the sample having to be re-digested.

9. Place vessel covers on top of vessel liners. Place a brown load disk on the vessel covers with the circular depression facing upward. For temperature/pressure control vessel, the load disk should have a hole in the center.
10. Install completed vessel assembly into support modules. Attach pressure tubing to the pressure port on the temperature/pressure control cover.
11. Rotate vessels so that the vent fittings are positioned toward the front of the support module (labeled end). With the module screw centered in the recessed area of the control cover, tighten the support screw finger tight. Using the torque wrench (preset at 5 ft-lbs), tighten the screw completely.
12. Turn on microwave by pushing switch at the back right of unit. Make sure vent hose is properly installed in hood.
13. Open microwave door using gray button on top. Place vessels in turntable slots 1-13.
14. Insert temperature sensor through module screw on control vessel. Slide the probe holder down the sensor and gently press it into the hole in the center of the module screw. Place the control vessel into turntable slot labeled "Control Vessel".
15. Snap the temperature sensor into connector at the top of the microwave cavity. Press the turntable key to rotate the turntable so the control vessel is positioned in the back left hand corner of the microwave.
16. Connect pressure cable into the port on the right side of the instrument cavity by rotating the connector while gently pushing it into the port until the polypropylene guard is full seated against the connector port. Place cable into holder on top of microwave cavity.

NOTE: Make sure pressure sensor cable does not pull temperature sensor up from control vessel. Press turntable key and watch to make sure sensor and cable are not twisting around one another.

17. Close microwave door.

18. Select appropriate method from the Windows software files.

The system is now ready to run.

1. After running a sample digestion, the vessels are allowed to cool for at least 5 minutes in the microwave.
2. Carefully unplug temperature and pressure sensors from the microwave and remove control vessel. Pull up on temperature probe holder to loosen. Carefully remove temperature sensor.
3. Remove all vessels and place in fume hood to allow further cooling. CAUTION: The sample vessels are very hot. If working with more than one tray, the next tray can be installed in the microwave while the first batch is cooling.
4. When vessels have cooled, carefully vent each vessel in the fume hood pointing the vent fitting towards the back of the hood. Remove the rupture disc and discard.
5. Loosen module screws with torque wrench and remove vessel assemblies from support modules. Remove load disks and covers.
6. Transfer samples to the labeled specimen cups and filter if necessary using Whatman #541 filter paper or equivalent.
7. Samples are ready for analysis.

NOTE: If sensor vessel should vent, sample volume will be lost and the whole set will need to be redigested. The PSI Guard will sound an alarm and shut down the unit.